

Patent
Attorney's Docket No. 032264-002

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Patent Application of

Thomas J. Taylor et al.

Application No.: 10/038,739

Filed: January 2, 2002

For: POLYCARBOXY/POLYOL
FIBERGLASS BINDER

Group Art Unit: 1713

Examiner: Judy M. Reddick

Confirmation No.: 3736

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DECLARATION PURSUANT TO 37 C.F.R. §1.132

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

Diana Fisler declares as follows:

1. That I am a citizen of the United States, and I reside at 2884 East Maplewood Avenue, Littleton, Colorado 80121.
2. That I have a Bachelor's degree in physics from the University of Massachusetts, 1987; and a Ph.D. in geophysics from Penn State University, 1994.
3. I have been working as a chemist for six years. My work has involved the chemistry of glass, and in particular, fiberglass, for the past five years, and I have been involved in the field of binder resins for fiberglass the past year.

(6/04)
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4. I have been employed at Johns Manville for the past six years as a chemist, working in the research of fiberglass and resins therefor. At present, I am the Binder Group Manager for Johns Manville, and have been for the past year.

5. I have reviewed the prior art cited by the U.S. Patent Examiner in the above-identified patent application, namely Arkens et al (U.S. Patent No. 5,427,587), Arkens et al (U.S. Patent No. 5,661,213), Arkens et al (U.S. Patent No. 5,763,524), Arkens et al (U.S. Patent No. 6,136,916), Chen et al (U.S. Patent No. 6,274,661 B1) and EP 583068 A1 (Arkens et al).

6. In the prior art, the molecular weight of the polymer employed in the binder resins can range broadly from 300 to 200,000 or more. Separately, the ratio of the hydroxyl/acid equivalents can range from 0.01 to 3. No connection is made between these two characteristics of the binder in the prior art.

7. The ratio of 0.01 to 3 for the equivalent for the hydroxyl to acid equivalence is a very wide range. At a ratio of greater than 1, for example 3, there would be excess polyol, which would be hazardous to consumers. It would also be very difficult to cure the binder resin, thereby yielding a low crosslink density, and a weak binder.

8. The prior art does disclose preferred ratios ranging from about 0.2 to 1, or even 0.2 to 0.8 hydroxyl/acid equivalents. These are also very wide ranges which encompass an exceedingly large amount of ratios and hence binder compositions, with the product quality varying enormously over the range. One working in the area of fiberglass binders would not consider these ranges as small, but would recognize

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each range as covering a large diverse number of compositions, particularly when one considers the combinations of a particular ratio with a molecular weight. There are many major changes in performance that occur throughout these ranges, many depending on and changing with the molecular weight of the polymer used, which is not even recognized in the prior art. For example, at a ratio of about 0.2, the crosslink density would be so low that a weak and non-durable binder is obtained. At a ratio of 0.5, with a low molecular weight polymer, i.e., less than 5000, an inferior binder with respect to crosslink density is achieved, whereas at a higher molecular weight, e.g., 10,000 or greater, a ratio of 0.45 to 0.50 is actually optimal.

9. There are certain examples in the prior art of low molecular weight, e.g., about 2000, but no examples of a low molecular weight polymer comprising binder also having a ratio of hydroxy/acid equivalence in the range of from 0.6 to 0.8. The commercial binder compositions generally contain a ratio of about 0.5, which as noted above provides an inferior binder with respect to crosslink density at low molecular weights, but is optimal for higher molecular weights.

10. It is surprising that the lower molecular weight polymer containing binders of the present invention requires a high polyol to acid ratio, thereby enabling better product performance and lower cost. The prior art does not recognize or suggest any important connection between the polymer molecular weight and the hydroxyl/acid equivalence ratio of the binder.

11. I further declare that all statements made herein of my own knowledge are true, and that all statements made on information and belief are believed to be

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true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under §1001 of Title 18 of the United States Code, and that such willful, false statements may jeopardize the validity of the application or any patent issuing thereon.

Dated: 11/18/04


Diana Fisler

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6. Under my supervision, binder mixtures comprised of polyacrylic acid (mw 4,000) and triethanolamine were prepared at different equivalent ratios of hydroxyl to acid groups. The binder mixtures were applied to a substrate of two rectangular pieces of Whatman fiberglass, sandwiched together. Approximately 0.26 grams of the desired formula was applied to the filter paper strips as uniformly as possible. The sample was then set up on a TA Instruments 2980 thermal mechanical dynamic analyzer using a dual cantilever clamp. A dynamic mechanical analyzer is an analytical tool for acquiring thermal data during the cure of a resin in question. The instrument is programmed to equilibrate the sample at 70°C, and then a temperature ramp of 4°C per minute is initiated until 270°C is reached. This temperature range was believed to sufficiently cover the needed heat to properly cure the samples of interest. All samples were adjusted to pH 3.1 with sulfuric acid, and all samples were prepared and analyzed by the same method. The dynamic mechanical analysis data used the initial and final extrapolated onset temperature, a

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tan delta, and a storage modulus. The samples prepared and tested are listed in the following Table 1:

Table 1

| <u>Sample No.</u> | <u>% TEA Equivalence</u> | <u>PAA (g)</u> | <u>TEA (g)</u> | <u>SHP</u> | <u>Water (g)</u> |
|-------------------|--------------------------|----------------|----------------|------------|------------------|
| 1 | 30% | 25.86 | 2.49 | 0.77 | 20.88 |
| 2 | 40% | 24.51 | 3.14 | 0.73 | 21.61 |
| 3 | 50% | 23.30 | 3.73 | 0.70 | 22.26 |
| 4 | 60% | 22.21 | 4.27 | 0.68 | 22.85 |
| 5 | 70% | 21.21 | 4.76 | 0.63 | 23.39 |
| 6 | 70% | 21.21 | 4.76 | 0.63 | 23.39 |
| 7 | 80% | 20.30 | 5.21 | 0.61 | 23.88 |
| 8 | 90% | 19.46 | 5.62 | 0.58 | 24.34 |
| 9 | 100% | 18.69 | 6 | 0.56 | 24.75 |

PAA – polyacrylic acid
 TEA – triethanolamine
 SHP – sodium hypophosphate

7. The results of the measurements is reflected in the following Table 2:

Table 2

| <u>Sample No.</u> | <u>% TEA Equivalence</u> | <u>T_g</u> | <u>T_f</u> | <u>Tan Delta @ 250°C</u> |
|-------------------|--------------------------|----------------------|----------------------|--------------------------|
| 1 | 30% | 141 | 165 | 0.060 |
| 2 | 40% | 144 | 170 | 0.046 |
| 3 | 50% | 145 | 173 | 0.036 |
| 4 | 60% | 148 | 177 | 0.032 |
| 5 | 70% | 154 | 183 | 0.028 |
| 6 | 70% | 153 | 182 | 0.029 |
| 7 | 80% | 153 | 184 | 0.027 |
| 8 | 90% | 154 | 186 | 0.035 |

| | | | | |
|---|------|-----|-----|-------|
| 9 | 100% | 161 | 193 | 0.045 |
|---|------|-----|-----|-------|

The results are also graphically illustrated in the appendix to this Declaration. The results show that the onset and final cure temperatures increase linearly with the triethanolamine level. The tan delta value is an indication of crosslink density. A more highly crosslinked binder may reduce product water absorption, wettability, and the transfer of moisture to the glass binder interface. The lower the tan delta value, the higher the crosslink density. The results indicate that for a hydroxyl/acid ratio between 0.6 and 0.8 (between 60% and 80% TEA equivalence) the best crosslink density is obtained for a low molecular weight polymer containing binder system. The optimum value was obtained at a ratio of 0.7 (70% TEA equivalence).

8. These results are quite surprising and unexpected, as prior binder systems have always employed a hydroxyl/acid equivalence ratio of about 0.5, which appears to be optimal for higher molecular weight binder systems. However, the above data surprisingly demonstrates that for lower molecular weight polymer containing binder systems, the crosslink density is surprisingly better at a ratio ranging from 0.6 to 0.8. Outside of this range, an inferior crosslink density is achieved for the lower molecular weight polymer containing binder systems.

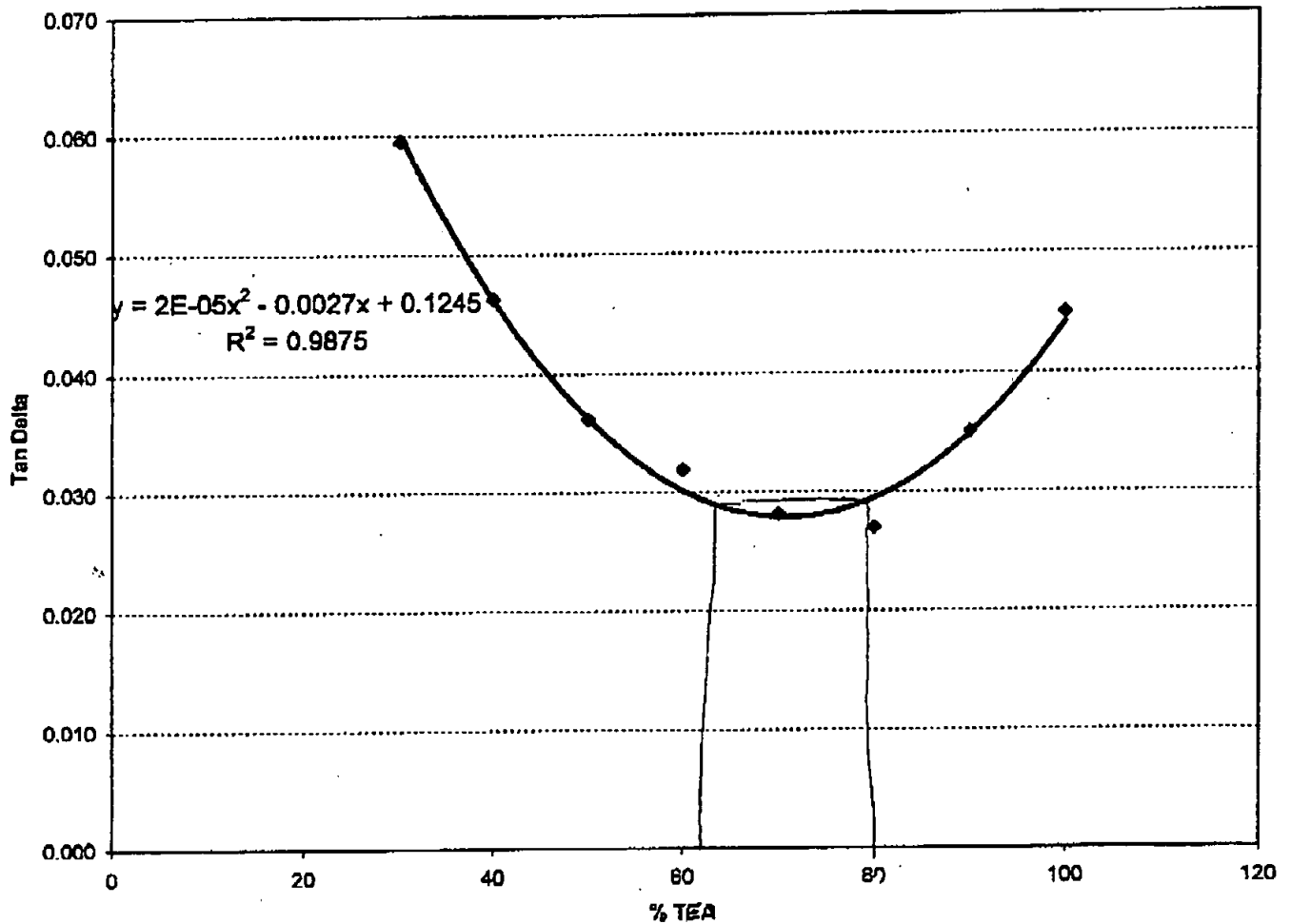
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9. I further declare that all statements made herein of my own knowledge are true, and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under §1001 of Title 18 of the United States Code, and that such willful, false statements may jeopardize the validity of the application or any patent issuing thereon.

Dated: 11/8/04


Diana Fisler

Chart Title



Diana Fister

Johns Manville
10100 W Ute Ave
Littleton, CO 80162-5005
Ph: 303 978 5296
Fax: 303 978 2214
Email fisterd@jm.com

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